

On Flue-dust and its
Composition as Obtained in
Roasting Argentiferous Ores

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"On flue-dust and its composition as obtained in roasting Argentiferous ores".

J. E. Weber,
for & me.



On Fluedust and its Composition as Obtained in the
Roasting of Argentiferous Ores.

In all establishments in which the roasting of ores is necessary either in the smelting industry or for the purpose of gathering the gases for the manufacture of sulfuric acid, there is always more or less trouble derived from the loss of ore and metal by escaping particles and "fume". This is the so-called Fluedust or "Chamber-dust" and may be defined thus: Fluedust consists of the small particles of the original ore which are mechanically dislodged, and of metals and their compounds that have been volatilized in the lower parts of the furnace and not condensed again in it, but carried along with the strong draft into the flue.

The volatilized metals or vapor of metals also go by the name of "fume"; The fluedust which results from the roasting of galena, pyrites, or other sulfur and argentiferous ores, has generally a dark reddish color, which is caused by the large percentage of iron oxide present and the presence of some particles of fuel in greater or less amount depending upon the manner of feeding the furnace, and upon the manner of stirring the fire. The amount of dust accumulated can be regulated to some extent. Careful

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feeding and cutting out of wall accretions will reduce the forming of fluedust to a great extent. High temperature in the smelting furnace causes much volatilization with consequent loss, and a strong blast will cause much of the vapor to be carried out of the furnace and into the flue. As the fluedust is derived from the roasting of Argentiferous and sulfur bearing ores, and since it contains a good percent of both metals and sulfur, it appears that there is a necessity for collecting the dust and fume in some way, so as to make it possible to recover the valuable metals contained in the ores.

Quite a number of methods for collecting the dust have been proposed.

The following methods seem to have produced the most satisfactory results. The flue leading from the furnace proper may be built so long and so large that the solid particles, they hold in suspension, will on account of their greater specific gravity drop out and accumulate on the floor of the flue. Such enlarged flues or chambers are now in use by large smelting concerns.

Then there may be modifications and improvements of these large chambers to further compel the retardation and cooling of the gases. A system of iron plates (Frendenberg plates) may be arranged in the roof of the flue longitudinally, that is parallel, with the current of the gas flow, together with a series of transverse

partitions built up from the floor to the edges of the ~~Fren~~denberg plates.

This arrangement will effectually arrest the flow of the gases and also cool them to some extent so that the fine dust will accumulate on the plates while the heavier particles will at once drop to the floor. The plates are suspended from 4 in. to 5 in. apart, and as the dust accumulates on them and has reached a thickness of about 1-1/8 in. to 1-1/2 in., it drops off automatically, or, by some contrivance, the plates may be shaken so as to free them from their dust load. This then accumulates in the compartments formed by the partitions on the floor.

The plates, will however, be corroded by the gases, and especially so, if there be any moisture present, because then both sulfurous and sulfuric acids are formed which will quickly corrode the plates. As these are expensive and their frequent renewal would be costly, a system of wires was devised by Roesing to take the place of the plates. The wires were arranged so as to form a screen running longitudinally with the flue, and by means of a shaking attachment, the dust would be much more easily dislodged from the wires than from the plates. As an example, of their efficiency might be cited the fact, that at "Farnowitz, Silesia, ✕ during 300 working days in 1888, there passed off into the open air without the wire system 121,000 lbs. of solid matter for every

furnace; with the wire system, the weight of the solid matter was reduced to 90,200 lbs. without diminishing the draft of the chimney."

But even though this arrangement is effective and its cost considerably less than that with plates, it is not entirely satisfactory, as being yet too expensive. So it seems that the most durable and least expensive dust chamber is an enlarged flue with transverse walls rising one or two feet above the floor to prevent the deposited dust from drifting along with the gas current. Between the upper edges of these walls and the roof of the chamber, there should be sufficient vertical space so as to cause the gases to flow slowly. The dust particles will then drop out in such a way as to leave the heavier and coarser ones, those most like the ore, near the furnace; as the particles are small and light, and highly volatilized they will settle farther and farther away from the furnace. This sorting out process is of great advantage when the accumulated dust is to be collected and resmelted.

In some smelters the gases are filtered through woolen bags, for the purpose of catching the fume. This process is inapplicable in the roasting furnace, on account of the sulfuric acid fumes, (SO₂) which are always present, and which would quickly destroy

any woolen bags that might be placed in the flue.

By means of the transverse compartments just mentioned and by having long chambers, the fume will be so cooled, that the principal portion of the metals contained in it, \times will have settled out before the gases are ready to enter the stack leading to the open air.

Thus will this important product be saved to the manufacturer, and can from time to time, be removed from the dust chamber through doors in the side walls.

After the dust has been collected, it is necessary to treat it in some way so as to make it possible to resmelt it. In the case of the sulfuric acid manufacturer, the dust is more of a nuisance than an important product, because the sulfur present in the pyrites, which he roasts, is almost all obtained in the first roasting; but yet he cannot disregard the dust as it contains important metals and compounds, which the smelter can use, and to whom it can be sold. Therefore in whatever concern there is an accumulation of fluedust, it will have to be prepared for its second smelting. The dust from smelting furnaces ^{sometimes} contains as much as 20% of the original ore. It would be poor economy, indeed, to let one-fifth of the original ore go to waste.

There seems to be considerable difficulty to treat the ~~dust~~ ^{dust} properly so as to make it possible to refeed it to the furnace. The best and favorite method proposed is to form or compact

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the fluedust into bricks. The dust is mixed with lime and zinc oxide, which when moistened, will make an adhesive mass, and then formed into bricks and hardened by drying, burning or freezing are added to the furnace charge.

Bricking the fluedust in this way makes a product that is easily handled, and one that will readily burn. Up till recent years the bricking was done by hand, but the modern plants form the fluedust into bricks by the aid of machinery.

The bricking presses have been able to reduce the cost from two collars per ton to from twenty to twenty-five cents per ton. Thus the fluedust as now treated and handled will save the manufacturer a good per cent annually, which would be a total loss, if it were not properly and carefully husbanded.

Having seen something of the source, accumulation, and methods of treating the fluedust for resmelting, it is the purpose of this paper to inquire somewhat into the composition of fluedust and especially into that which is derived from the sulfuric acid works at Argentine, Kansas. In doing this it seems necessary to go somewhat into the details of the processes of the manufacture of sulfuric acid.

The process used at Argentine is the common lead-chamber process. The iron pyrites are roasted, in Herreschoff Roasters, of which there are five pairs. The ore is dropped into the hopper of the furnaces from the floor above, and the hoppers feed the ore to

the roasters automatically. The roaster is built up so as to contain a number of compartments, one above the other, and a verticle shaft runs through the center of each roaster. To this central shaft, which is turned by an engine, is attached toothed arms, or rakes, two in each compartment, and the teeth of these rakes are so arranged that the ore is gradually moved towards the center in one compartment, and then drops through holes down into the next compartment. Here the rakes have their teeth set at such an angle that the ore is moved outwards toward the circumference, and there drops through holes to the next lower compartment, and so on for six or seven stories until after eight or ten hours, the ore has been so thoroughly heated and stirred that almost all the sulfur has been roasted out. Only 5% to 2% remain in the ~~ore~~ *cinder*.

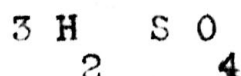
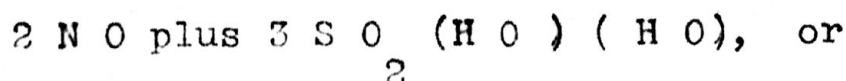
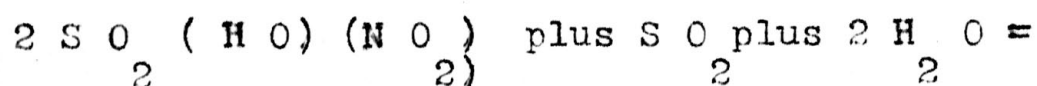
The supply of atmospheric oxygen is regulated by a system of draft holes. The hot sulfur dioxid formed by the burning sulfur of the pyrites, enters a flue common to the ten roasters. Near the end of this flue is the "nitre pot", an arrangement for producing the nitrogen gases, which is heated by the flowing sulfur dioxid and oxygen. All these gases then mix and enter the large room, which is built of brick and is provided with transverse brick walls rising from the floor.

This is the dust chamber or flue, from which this particular sample of dust was collected. As the gases from the roasters enter this chamber their flow is retarded and the particles mechanically carried along are deposited in the floor compartments.

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Having traversed the dust chamber, the gases which consist of sulfur dioxid, ¹nitrogen and oxygen, are made to pass up the so-called Glover Tower.

This is a large, leaden tower, packed full of small pieces of coke. As the gases pass up the tower they meet a stream of nitro-sulfonic acid which has been previously formed in the ~~Gay~~^{Gay}-Lussac Tower. The effect of the gases on the dilute sulfonic acid is to remove a portion of the water from it, and thereby effect a partial concentration. The gases also cause the denitrification of the nitro~~s~~ sulfonic acid thus:



From the Glover Tower the gases are delivered into a series of large lead lined chambers, where they come in contact with steam. The acid formed falls to the bottom of the chambers, and samples of the acid is frequently drawn off and examined by an arrangement known as the " drip-pipe". After the gases have been drawn through the entire system of leaden chambers, by means of the draught caused by a tall chimney, they are finally passed up the ~~Gay~~^{Gay} Lussac tower.

this consists of a square leaden tower filled with fragments of coke, into the top of which is admitted, and evenly distributed, by means of the Segner wheel, cold, strong sulfuric acid, which percolates slowly through the mass of coke.

The gases meeting this stream of cold sulfuric acid, lose all their nitrogen peroxide and form with it nitrosulfonic acid. ~~(SO₂)~~.

This is then pumped up into the top of the Glover tower, and the cycle is completed. The acid thus formed is dilute and is subsequently concentrated in glass or platinum retorts.

There is coming into vogue another method of manufacturing sulfuric acid which is called the contact process. This is essentially a process which makes use of the catalytic action of *heated* platinum, of the oxids of iron, copper, and chromium in causing SO₂ and O to unite to form S O₃ which can afterwards be chemically united with water to form H₂ S O₄. It has the advantage of doing away with the expensive lead chamber system, the Glover and Gay-Lussac towers, and the plants for producing steam and nitric acid. On the other hand, it will require a well planned and carefully managed system for the purification by the roaster gases; and will also need a high order of engineering skill to run it successfully. Nevertheless, there seems to be good reasons for believing that the contact process will in the near future mostly, if not entirely, replace the old leaden chamber process.

The sample of fluedust in hand was, as stated, above, obtained

from the flue of the sulfuric acid plant at Argentine.

In order to determine its composition, a qualitative examination was first made of the dust. Five grams of a sample, obtained by careful mixing and sampling of the whole amount of dust at hand, were treated to nitrohydrochloric acid repeatedly until nothing more would dissolve. The insoluble residue was filtered off, and the solution evaporated nearly to dryness in order to get rid of as much acid as possible. Diluted the thick, reddish brown substance with water, added some hydrochloric acid and treated the solution with hydrogen sulfid gas.

The precipitate was washed thoroughly with hot water and ammonium acetate. It was put into a solution of yellow ammonium sulfid to which was added some ammonium hydroxid and warmed for some time, after which the liquid was decanted off, and the process repeated, four times. This dissolved nearly all of the precipitated sulfids with the exception of a small residue, which was filtered off.

The filtrate was treated with dilute hydrochloric acid, which reprecipitated the sulfids of arsenic, antimony, and tin. They were now heated without boiling in concentrated hydrochloric acid. This process dissolved nearly all of the precipitate, except the arsenic and some sulfur. The mixture was then treated to some potassium chlorate and heated until red litmus was not bleached. It was then tested for arsenic, antimony and tin by treating it with an abundance of concentrated hydrochloric acid and hydrogen sulfid. This treatment

brought out the arsenic sulfid, and by further treating it with a solution of hydrogen sulfid, the antimony sulfid was precipitated. The stannic sulfid appeared to be there, but was very indistinct.

The metals, arsenic and antimony, were decidedly present with a possibility of tin being present in small quantity.

During the precipitation of the sulfids with H_2S , there seemed to be no end to the reaction, because a lemon yellow precipitate kept coming down continuously after every filtration and a whole week was consumed in throwing down the sulfids. This precipitate was afterwards proven to be nothing but sulfur, although, at first, thought to be Selenium. The lemon yellow precipitate would not stand any of the tests for selenium with the one exception that it was soluble in yellow ammonium sulfid. After it was isolated it was found to dissolve in CS_2 . The probable reason for the sulfur coming down so plentifully and of a lemon yellow color, might be ascribed to the great abundance of iron present in the solution, which would have the effect of decomposing the hydrogen sulfid and setting free the sulfur.

The undissolved residue by yellow ammonium sulfid, was now treated to dilute nitric acid and allowed to simmer for a short time. There appeared no precipitate so that mercury was found to be absent. Dilute sulfuric acid was then added to the nitric acid solution and evaporated until sulfuric acid fumes (SO_3) were abundantly given off, and a white precipitate of lead sulfate was

thrown down. It was confirmed by treating the precipitate with sodium hydroxid which dissolved the lead sulfate.

Acidifying this alkaline solution with acetic acid and adding potassium bichromate, the yellow precipitate of lead chromate was thrown down, proving the presence of lead.

The filtrate, from which the lead sulfate was filtered off, was now treated with ammonium hydroxid and heated to boiling, but no precipitate of any kind was thrown down, showing the absence of bismuth. The alkaline solution was of a very faint blue color, showing the presence of copper in minute quantity. When a small portion of it was treated with acetic acid and potassium ferrocyanid the well known reddish brown precipitate of cupric ferrocyanid was formed. The greater portion of the alkaline solution was acidified with hydrochloric acid and evaporated down to small volume; it was then saturated with pure sodium chloride and an abundance of hydrogen sulfid was added. The solution was then filtered, and the filtrate was diluted very greatly with water and made alkaline with ammonium hydroxid and hydrogen sulfid added. The absence of cadmium was proven by the characteristic yellow precipitate of cadmium sulfid failing to come down.

The filtrate from the precipitated sulfids was boiled with some nitric acid to fully oxidize the iron, after which it was treated to ammonium hydroxid to strong alkaline reaction.

A very large precipitate was obtained, which was filtered off. The filtrate was reserved for further tests.

The precipitate was thoroughly washed with hot water and treated to about six grams of sodium peroxid and boiled. Filtered off the iron hydroxid and divided the filtrate into two parts. To one portion was added acetic acid in excess and lead acetate, when the characteristic yellow precipitate of lead chromate was obtained thus proving the presence of chromium. To the second portion was added an abundance of ammonium chlorid and boiled until all ammonia was driven off; then more ammonium chlorid was added, and the solution boiled again, a flocculent white precipitate insoluble in water was obtained, which indicated the presence of aluminum. A portion of the iron hydroxid was dissolved in H Cl. and ^{tested} (for iron with potassium ferrocyanid and potassium sulfocyanid, both of which tests proved the presence of iron. Another small portion was fused with sodium carbonate and potassium nitrate in a porcelain crucible, but the iron present was so abundant as to completely obscure any green coloration that may have been formed by the presence of manganese.

Later, manganese was found to be present in a fusion from which the greater part of the iron had been removed, but there was at most only a mere trace of manganese present.

The filtrate from the hydroxids was treated to ammonium sulfid which brought down a precipitate. This was treated with cold dilute

hydrochloric acid to dissolve the sulfids of managanese and zinc. Since there was practically no residue there was no nickel or cobalt present. The filter and solution were both tested for cobalt and nickel with the borax bead, but no coloration indicated the absence of both of these metals.

The filtrate was tested for zinc by adding to it a clear solution of sodium acetate saturated with hydrogen sulfid, until precipitation was complete. The white precipitate showed the presence of zinc.

The filtrate from the treatment with ammonium sulrid was treated to ammonium chlorid and ammonia and warmed, after which a solution of ammonium carbonate was added. No precipitate appeared thus proving the absence of alkali earth metals. This practically completed the qualitative analysis of the dust.

For the quantitative determination of the various components of the dust, two grams were taken, and the analysis was done in duplicate. The dust was dissolved in aqua regia as thoroughly as possible, and the liquid was carefully decanted off. This process was repeated four times. The liquid was diluted with some water and filtered. The insoluble residue was dried, ignited and weighed with the following results:

Weight of crucible and ppt.	16.5227 g.	14.4679g
	2	31
Weight of " and Ash,	<u>16.4170 g.</u>	<u>14.3629g</u>
Weight of insoluble residue	0.10 55g.	0.1048g

This insoluble residue was then fused in platinum crucibles with sodium carbonate, and a little potassium nitrate for the determination of silica. After the silica was filtered off the resulting filtrate was added to the aqua regia filtrate. The results from this treatment were:

(1)

Weight of crucible and ppt.	14.9844	=	15.6994
" " " "	Ash, <u>14.8786</u>		<u>15.5947</u>
Weight of silica	0.1056g.		0.1045 g

which gives a percentage of 5.28~~8~~ %

5.23~~8~~ %

Now one half gram of the original dust was fused with sodium carbonate and a little potassium nitrate in platinum crucible as a check on the correctness of the percentage silica, when the following results were obtained:

(2)

Wt. of crucible & ppt.	6.1547	6.9635
Wt. " " & Ash,	<u>6.1083</u>	<u>6.9364</u>
Wt. of Silica	0.0262	0.0269

Which gives the per cent 5.24% and 5.38%.

Thus it seems that the average of the first set of results, viz: 5.25%, would be a safe estimate of the amount of silica present.

The aqua regia solution together with the filtrate from the fusion of the insoluble residue (not that from the 5 g. determination) was evaporated down to a very small bulk for the purpose of getting rid of as much nitric acid as possible. Then it

was diluted considerably with water and a little hydrochloric acid added. Hydrogen sulfid was now added until a colored precipitate ceased to come down. This precipitate was treated to ammonium hydroxid and yellow ammonium sulfid and warmed for some time.

Then it was diluted considerably with water and a little hydrochloric acid added. Hydrogen sulfid was now added until a colored precipitate ceased to come down. This precipitate was treated to ammonium hydroxid and yellow ammonium sulfid, and warmed for some time.

This operation was repeated four times. The insoluble residue was dissolved in aqua regia and evaporated somewhat. It was then diluted with water and dilute sulfuric acid was added to precipitate the lead. Then a considerable quantity of alcohol was added to the solution, and it was left standing for some time. The precipitated ^{lead} sulfate was then filtered off and thoroughly washed with alcohol, dried, heated and weighed, with the following results:

	(1)	(2)
Wt. of crucible & ppt.	6.7074 g	5.9999g
" " " & Ash	<u>6.6700 g</u>	<u>5.9610 g</u>
Weight of PbSO ₄ =	.0372 g	.0387g

Which gives the percent of lead 1.28 % 1.32 %

" " " " "PbO 2.05 %

The filtrate from the lead sulfate was treated to some more sulfuric acid and evaporated to dryness. This gave a very small

precipitate of copper sulfate.

This was dissolved in water and neutralized with sodium carbonate until a permanent precipitate appeared. This was dissolved in an excess of acetic acid. About one half gram of potassium iodide was added, and the solution thus prepared was titrated with standard sodium tetrathionate sulfate solution. $\text{Na}_2\text{S}_4\text{O}_6$ whose factor was $1 \text{ c.c. } \text{Na}_2\text{S}_4\text{O}_6 = 0.00474 \text{ g. Cu.}$ of which the copper solution required one half c.c. in one test and very nearly the same, possibly .47 c.c., in the duplicate test.

The result for copper was then $.5 \text{ c.c. } \times 0.00474 = .00237 \text{ g. copper or } 12\% \text{ Cu. or } 0.15\% \text{ Cu.O. in the dust.}$

The solution of sulfids, in yellow ammonium sulfid, was diluted up to one liter or 1000 c.c. .500 c.c. of this solution was taken for the determination of arsenic. The sulfids were first reprecipitated with dilute H Cl.. Then digested with sulfurous acid and potassium bisulfate (since potassium sulfite could not be obtained). It was then boiled until all of the sulfurous acid was expelled, and then filtered.

The arsenic was now all in the filtrate, and was mixed with ammonia in excess. Magnesia mixture was added, and the solution, which smelled strongly of ammonia, was covered and left standing for two days. The precipitate was transferred to a filter, thoroughly washed, with a dilute solution of ammonia; it was then dried in the oven. When dry, as much as possible of the precipitate

was removed from the filter, which was saturated with a solution of ammonium nitrate and dried. Then carefully burned in a porcelain crucible. After cooling, the precipitate was added to the ash, heated at first gently, then with greater heat, for four hours. The following results were obtained:

	(1)	(2)
Wt. of crucible and ppt.	6.1184 g	6.9820 g
" " " " Ash	<u>6.0865</u>	<u>6.9510</u>
Wt. of Mg 2 As 2 O	0.0317 g	0.0308 g
Wt. of As. = $\frac{150 \times 0.0317}{310}$	= .0153 g	= $\frac{150 \times 0.0308}{310}$ = 0.149g

That is in 500 c c; in one liter there would therefore be .0153 X 2 = .0306 g; .0149X2= .0298g.

Which gives the percent for 2 grams = 1.53% and 1.49%.

For the determination of antimony 250cc of the yellow ammonium solution was treated with hydrochloric acid.

The precipitate was allowed to settle for some time. The greater portion of the superatant liquid was carefully decanted off.

The precipitate was now treated with concentrated hydrochloric acid and carefully heated up to 70°. The solution, thus prepared, was diluted with water and kept acid with hydrochloric acid, then titrated with standard potassium-permanganate solution, whose factor was 1 c c K Mn O 4 = .00663g. *Sb.*

S. Each test solution required 4.5c c K Mn O 4, which therefore gave 4.5 X .00663 = .0285g. *Sb.* .0285X4 = .1140g *Sb.* or $\frac{1140 \times 100}{2} =$

5.70 % *Sb.* The remaining 250 cc were treated to an abundance

of oxalic acid according to Fresenius, but there was not a sign of tin obtained.

The filtrate obtained from the sulfids precipitated with H_2S was diluted up to 500 c c. Of these, 100 c c were taken for the determination of zinc. The solution was treated with nitric acid, boiled down to small volume, and then potassium chlorate was added. The mixture evaporated to dryness and heated for some time.

Extracted the zinc with ammonia and ammonium chlorid solution, boiled, filtered and washed with dilute extraction solution. Added HCl in excess, heated the filtrate to boiling and titrated hot with standard potassium ferrocyanid, whose factor was $10 \text{ c c } K_4Fe(CN)_6 = .00959 \text{ g Zn}$. Of this the test solution required 2 cc which multiplied by 5 gives a total of 10 c c of the potassium ferrocyanid solution required. Hence there was present $10 \times .00959 = .0959 \text{ g. Zinc}$ or $\frac{.0959 \times 100}{2} = 4.78\%$ zinc, or $5.92 \% \text{ Zn.O}$.

For the determination of iron 50 c c of the solution *and evaporated until dense sol.* were treated with sulfuric acid fumes were given off. It was then diluted and reduced with H_2S . The excess of H_2S was completely boiled off, the solution cooled, keeping the mouth of the flask closed as much as possible, some H_2SO_4 added, and titrated with $KMnO_4$, whose factor was $1 \text{ c c } KMnO_4 = .00533 \text{ g. Fe}$. Of this the test solution required 12.2 c c $KMnO_4$. Hence there was found present $12.2 \times .00533 \times 10 = .6502 \text{ g}$.

$$\text{Fe.} = 32.51 \% \text{ Fe. and } \frac{32.51 \times 160}{112} = 46.44\% \text{ Fe. } \begin{matrix} 0 \\ 2 \end{matrix} \begin{matrix} 3. \end{matrix}$$

The Chromium was determined by taking 100 c c of the solution and precipitating with ammonium hydroxid. This was washed, dried, heated and weighed with the following results:

Weight of crucible and ppt. 16.6234 g

" " " ash 16.4200 g

" " Fe." $\begin{matrix} 0 \\ 2 \end{matrix}$ $\begin{matrix} 3 \end{matrix}$ Al $\begin{matrix} 0 \\ 2 \end{matrix}$ $\begin{matrix} 3 \end{matrix}$, Cr. $\begin{matrix} 0 \\ 2 \end{matrix}$ $\begin{matrix} 3 \end{matrix}$ = .2032 g. = $\frac{.20.32 \times 100}{.4} = 50.80\%$

This dried precipitate was now carefully pulverized and fused with four grams sodium carbonate and a little potassium nitrate. The mass was then boiled and some alcohol added, when the mixture was allowed to heat gently for five hours. It was then filtered and the filtrate treated with potassium chlorate, acidified with hydrochloric acid and evaporated to the consistency of a thick syrup; adding some more potassium chlorate as the evaporation went on. The solution was diluted with water, and the aluminum precipitated with ammonia and boiled, filtered and washed. Now was added sodium acetate in excess and acetic acid until strongly acid, after which the chromium was precipitated as lead chromate. It was dried in the oven, carefully heated over the bunsen burner, and weighed, with the following results:

Weight of crucible and ppt. 14.65379
 " " " " ash, $\frac{14.2339}{41}$
 " " Pb. O_2 ----- 0.3196

$$\text{Weight of Cr}_2\text{O}_3 = \frac{152 \times 3196}{646} = .0752 \text{ g. Cr}_2\text{O}_3$$

Hence there was found to be present

$$0.0752 \text{ g} \div .4 \text{ g} = 1.88 \% \text{ Cr}_2\text{O}_3 \text{ or } 1.29 \% \text{ Cr.}$$

Now, since the Fe. O_3 , Cr. O_3 ~~and~~ and Al O_3

together represent 50.80 % and the Fe. $\text{O}_3 = 46.44 \%$ and Cr. O_3

= 1.88, it was possible to determine the aluminum by difference thus

$$50.80\% - (46.44 \text{ plus } 1.88) = 2.48\% \text{ Al}_2\text{O}_3 = 1.31\% \text{ Al.}$$

In order to determine the sulfur, one gram of the dust was treated repeatedly with aqua regia, then all the decantations and residue were evaporated almost to dryness. Diluted with water and filtered off the insoluble residue. Added a little H Cl. and hot barium chlorid until precipitation was completed. The mixture was allowed to stand for several hours. The precipitate was filtered off, dried, burned, and weighed.

Weight of crucible and ppt. 15.0700g

" " " " ash, $\frac{14.3750}{2}$ g

" of Ba S O 4 = .6948 g

$$\text{Wt. of S O}_3 = \frac{.6948 \times 80}{233} = .2386 \text{ g S O}_3 \text{ or } 23.86 \%$$

The silver and gold was determined by fire assay, with the following charge:

Fluedust $1/2$ assay ton.

Lead Flux 40 g

Na H C O $\frac{20}{3}$ g

Litharge 25 g

Silica 15 g

Nails, 3

Borax cover.

The two silver buttons weighed $8-1/3$ mg. and $8-8/9$ mg. respectively, which makes an average of about 17.33 oz. of silver to the ton. There was a distinct trace of gold but in too minute a quantity to turn the balance.

The alkalies, if any, and the moisture were undetermined. Through the kindness of Prof. Cady, the dust was tested for radio-activity, by an instrument which consists essentially of an extremely sensitive electrometer. But it showed not the least sign of radio-activity and hence proved the absence of uranium.

Summary of the Analysis.

Si O ₂	5.25 %	5.25 %	Si O ₂
Pb.	1.28%	2.05%	Pb O
As.	1.52%	2.01%	As ₂ O ₃
Sb.	5.70%	6.84%	Sb ₂ O ₃
Cu.	.12%	.15%	Cu ₂ O
Zn	4.79%	5.92%	Zn O.
Fe	32.51%	46.44%	Fe ₂ O ₃
Cr	1.29%	1.88%	Cr ₂ O ₃
Al	1.31%	2.48%	Al ₂ O ₃
S.	9.44%	23.86%	S O ₃
Ag. Oz. per ton	17.33		
Au. " " "	Trace		
Alkalies	Undetermined		
H ₂ O.	Total substance <i>percent</i> -- 96.88%		

Authorities consulted in the preparation of this paper.

Qualitative Analysis: Fresenius. Bailey and Gady,
Prescott and Johnson, Newth,

Quantitative
~~Qualitative~~ Analysis: Fresenius, Newth, Cairnes.

General Chemistry: Ostwald, Newth.

Assaying: Furman.

Metallurgy of Lead: Greenwood, Eisslar, Hofmann

iles, Sexton, ~~Ingalls~~ Ingalls.

Manufacture of Sulfuric acid: A. G. and C.G. Lock.

Bulletin 12th Census.
on Chemicals.

Rare metals: Dr. J. Ohly,

Prof. Bailey has always throughout the whole course of the analysis cheerfully given advice and encouragement.

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